Original document

PROCESS FOR PRODUCING HIGH-STRENGTH SEAMLESS STEEL PIPES EXCELLENT IN SULFIDE STRESS CORROSION CRACKING RESISTANCE

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Abstract of corresponding document: **EP0224591**

A process for producing seamless steel pipes to be used as oil well pipes, line pipes, piping for chemic plants, etc. having both high strength and high sulfide stress corrosion cracking resistance by using inexpensive components, which process comprises increasing the content of C and minimizing the contents of impurities such as P and S, conducting quenching at a relatively slow rate of 1 to 50 DEG C/s after heat treatment, then tempering in a relatively high temperature range of from 680 DEG C to the Ac1 transformation point. The pipe thus obtained has a yield strength of 75 kgf/mm<2> or more ar excellent stress corrosion cracking resistance without causing any quenching crack.

Description of corresponding document: **EP0224591**

Specification
PRODUCTION OF HIGH STRENGTH SEAMLESS
STEEL PIPE WITH SUPERIOR RESISTANCE
AGAINST SULFIDE STRESS CORROSION CRACKING
Technical Field
The present invention relates to a method of producing a high strength seamless steel pipe with a superi resistance against sulfide stress corrosion cracking. The steel pipe is favorably used for oil well pipes un souring tendency and extremely deepening well, line pipes for sour gas and sour oil, chemical plant pipe

lines, etc. The invention discloses development outcomes to effectively avoid dangers of occurrence of sulfide stress corrosion cracks under a high yield strength of at least 75 kgf/mm2.

Technical Background
Since the sulfide stress corrosion crackingresistance is generally deteriorated with increase in strength, (Mo base steel having a yield strength of around 64 to 74 kgf/mm2 has been to date considered to be mo: excellent against the above-mentioned oil well deepening and souring tendency to attain both the properties in combination.

According to Japanese patent application laid-open No. 53-78,917, there has been recently developed a steel having about 75 to 90 kgf/mm2 aiming at improvement on the sulfide stress corrosion crackingresistance through increasing Cr andMo and adding a larger amount of V as compared with a conventional
Cr-Mo steel having around 65 kgf/mm2. However, such a steel is expensive because it contains large amounts of expensive elements such asMo and V. Further, since a large amount of V is contained, the st is likely to develop cracks during hot processing in a continuous casting.

Japanese patent application laid-open
Nos. 57-19,322 and 57-19,323 proposed La-added steels excellent in sulfide stress corrosion cracking-resistance, but the yield strength of these steels is around 80 kgf/mm2 at the maximum. Therefore the strength is hardly improved when compared with the conventional steels.

Furthermore, Japanese patent application laid-open No. 57-35,622 discloses a high strength oil well stee which P and S are reduced. However, although the strength of this steel is considerably improved, the stress corrosion cracking-resistance is assured only when H2S is contained in a small amount under an alkaline environment.

Moreover, Japanese patent application laid-open
Nos. 52-52,114 and 54-119,324 propose steels in which the sulfide stress corrosion cracking-resistance : improved through making steel grains finer by performing heating at the average heating rate of 3 to 500C/s in a range of Ac1 transformation point or higher (Japanese patent application laid-open No. 52-52,114) or rapidly performing cooling at about from 1 to 500C/s (Japanese patent application laid-open No. 54-119,324) for quenching. However, such processes have limit in attaining both high strength and sulfide stress corrosion cracking-resistance in combination.

In practice, the yield strength of the steel disclosed in Japanese patent appliation laid-open
No. 54-119,324 is about 65 kgf/mm2, while in the case of the steels disclosed in Japanese patent application laid-open No. 52-52,114, the maximum load stress at which no sulfide stress corrosion cracl occur is only 55 kgf/mm2 (73% of the yield strength) when the yield strength is 75 kgf/mm2 and only about 40 kgf/mm2 (44% of the yield strength) when the yield strength is 90 kgf/mm2.

Under such current circumstances, there has been recently increased damand for high strength materials having a yield strength of not less than 75 kgf/mm2 and an excellent sulfide stress corrosion cracking-resistance in combination, and seamless pipes of an Ni base alloy such as Hastelloy, or inconel have bee tried to be used as oil well pipes. However, since such materials are too expensive, use thereof is oblige be restricted. Therefore, development of inexpensive low alloy steel base materials have been strongly demanded.

Disclosure of the Invention
An object of the present invention is to advantageously solve the above-mentioned problems, and to provide a process for advantageously producing a seamless steel pipe which is composed of inexpensive

ingredients suitable for continuous casting without containing a large amount of the above-mentioned expensive elements and which has an excellent sulfide stress corrosion cracking-resistance and a high yi strength of from 75 to 120 kgf/mm2 in combination.

In order to solve the above-discussed problems, the present inventors have acquired the below-mentione acknowledgement through repeatedly strenuous researches.

(1) While the sulfide stress corrosion cracks are more likely to occur with increase in strength, the higher an annealing temperature, the more excellent the sulfide stress corrosion cracking resistance in the case of the same level strength.

(2) In order to obtain superior sulfide stress corrosion cracking-resistance in the case of the steels having a yield strength of not less than 75 kgf/mm2, it is necessary that the annealing temperature is set at not less than6800C.

(3) In order to stably obtain a yield strength of not less than 75 kgf/mm2 when the annealing temperature is set at not less than 6800C, it is necessary that Cr and Mo are not less than 0.8% by weight (hereinafter referred to briefly as and 0.6%, respectively, in the case of a Cr-Mo steel containing more than 0.40% of C.

(4) When P is present at not less than 0.020% or S is present at not less than 0.010% in the steel, excellent sulfide stress corrosion crackingresistance can be obtained even when the annealing temperature is set at not less than680"C.

(5) As compared with a steel having strength improved by Cr and Mo only as stated in the above item (: a steel further admixed with at least one of Ti, V and Nb is improved in terms of the sulfide stress corrosion cracking-resistance.

In particular, a sour resistance critical stress ratioreaches 90% or more by adopting a Cr-Mo-Ti-V-Nb system. Here, the sour resistance critical stress ratio is a ratio expressed by percentage between the maximum stress at which no sulfide stress corrosion cracks occur at a souring environment and the yielc strength of a material.

(6) However, when Cr-Mo steel containing as much as over 40% of C as intended in the present inventi is subjected to a quenching treatment in a form of a long and thick pipe such as an oil well pipe, quench cracks occur when it is merely put into a cooling medium such as water or oil from a heated state as ordinarily done.

Therefore, it is necessary to perform cooling relatively slowly. However, when the cooling rate is too small, no quenching is effectuated and the sulfide stress corrosion cracking-resistance after the annealing is remarkably deteriorated.

In order to prevent this, it is necessary that 90% or more of the structure after the quenching are convert

to martensite. For attaining this, it is necessary that the average cooling rate is set at not less than10cos i range from the heating temperature to around 3000C at which the martensite transformation is almost completed. On the other hand, in order to prevent the quenching cracks, it is necessary to set the average cooling rate at not more than 500C/s.

The present invention is based on the abovementioned acknowledgement.

That is, the present invention relates to a process for manufacturing a high strength seamless steel pipe having an excellent sulfide stress corrosion cracking-resistance, which process comprises hot processing hollow raw material for the seamless steel pipe, heating the hot processed raw material up to a temperati of not less than an Ac3 transformation point, quenching the resultant while cooling at the average coolir rate of from 1 to 500C/s is done in a range from the Ac3 transformation point to at least 3000C, thereby making conversion into martensite structure, and then annealing a quenched article in a temperature ran; of from not less than 6800C to not more than the Ac3 trans formation point, said raw material containin; from more than 40 to 0.60% of C, from 0.20 to 0.35% of Si, from 0.4 to 1.2% of Mn, from 0.8 to1.5% o Cr, from 0.6 to 1.0% of Mo, and from 0.005 to 0.1% of Al together with P and S being restricted to not more than 0.020% and not more than 0.010%, respectively, and occasionally further at least one kind of V and Nb in a range of not more than 0.1%.

In the following, the present invention will be concretely explained.

First, the reasons why the components of the raw material are restricted to the respective above ranges according to the present invention will be explained below.

C : more than 0.40 to 0.60%
C is an element useful for obtaining a high strength in quenching even at high temperatures.

In particular, in order to stably obtain a yield strength of not less than 75 kgf/mm2 through annealing at high temperatures of not less than 6800C as intended by the present invention, at least more than 0.40% C is necessary. If it exceeds 0.60%, there is possibility that quenching cracks occur. Thus, C is added in range from more than 0.40 to 0.60%.

Si : 0.20 to 0.35%
At least 0.20% of Si is necessary to improve deoxidation and strength of the steel. If it exceeds 0.35%, t toughness is deteriorated. Thus, Si is restricted to a range from 0.20 to 0.35%.

Mn : 0.4 to1.2%
Mn is an element useful for improving hardenability and strength as well as for deoxidizing.

If the content is less than 0.4%, effect obtained by the addition is poor, while if it exceeds1.2%, it cause: segregation of P, S, etc. to deteriorate the sulfide stress corrosion cracking-resistance. Thus, Mn is inclu in a range from 0.4 to 1.2%.

Cr : 0.8 to 1.5%
Cr forms carbides during quenching and annealing treatment to effectively contribute to increase strengt and annealing resistance. For this purpose, it is necessary that 0.8% or more of Cr is added.

If it is added in an amount of more than 1.5%, its effect is not only saturated, but also the sulfide stress corrosion cracking-resistance is inversely deteriorated. Thus, Cr is restricted to a range from 0.8 to1.5%

Mo :0.6. to 1.0%

As in the case of Cr, Mo increases strength and resistance against annealing and further effectively contributes to the improvement of the sulfide stress corrosion cracking-resistance through preventing th segregation of P in grain boundaries. If the content is less than0.6%, effect obtained through the additio poor. On the other hand, even if it is included at more than 1.0%, its effect is not only saturated, but alsc toughness tends to be inversely deteriorated and cost rises. Thus, Mo is restricted to a range from 0.6 to 1.0%.

Al : 0.005 to 0.1%
Al is a useful element which not only contributes to deoxidation but also improves toughness, strength a sulfide stress corrosion cracking-resistance through making crystal grains fine upon reaction with N.

However, if the content is less than0.005%, the effect obtained through the addition is poor, while if it i over 0.1%, the effect is not only saturated, but also the toughness is inversely deteriorated. Thus, the content is restricted to 0.005 to0.1%.

P : not more than 0.20%; S : not more than 0.010%
Both P and S are harmful elements which cause conspicuous deterioration of the sulfide stress corrosion cracking-resistance of the steel. In order to maintain the intended strength and sulfide stress corrosion cracking-resistance when Mo is added and the annealing temperature is set at a high temperature of 680 or more according to the present invention, it is necessary that P and S are restrained to not more than 0.020% and not more than 0.010%, respectively.

A molten steel formulated in the above-mentioned preferable ingredient composition is cast, and conver to a hollow material through piercing according to a conventional way.

The thus obtained hollow material for a seamless steel pipe is hot rolled, and then quenched and anneale

It is necessary to convert 90% or more of the steel structure to martensite structure during the quenching without causing quenching cracks. For attaining this, it is necessary that after the steel is heated at temperature of not less than Ac3 transformation point, it is cooled at the average cooling rate of from 1 t 500C/s, preferably from 5 to 300C in a range from the Ac3 transformation point to at least 3000C.

The reason why the average cooling rate is restricted to the range from 1 to 500C/s is that if the average cooling rate is slower than1OC/s, the steel is not sufficiently quenched and therefore satisfactory sulfide stress corrosion cracking-resistance after the annealing cannot be obtained, while if it exceeds 500C/s, there is a great possibility that quenching cracks occur.

As a cooling way, various methods are available, for instance, a method of applying a cooling medium t the steel pipe through spraying, showering, fogging, or the like while an amount of the cooling medium, the application pressure, etc. are controlled, a method of cooling the steel pipe with water or oil after the heating in the state that the pipe is preliminarily wrapped with an excellent thermal resistant and excelle temperature-maintaining refactory material prior to the heating, or a method of formulating such a cooli medium as to meet the intended cooling capability.

Any method may be adapted so long as the average cooling rate in the above range is satisfied.

It is necessary to perform annealing treatment at a temperature range of not less than 6800C but not mo than the Ac1 transformation point. For, in order to obtain the yield strength of from 75 to 120 kgf/mm2 the excellent sulfide stress corrosion crackingresistance as intended by the present invention, it is indispensable to perform the annealing treatment at a high temperature of not less than 6800C. On the other hand, if the temperature exceeds the Ac1 transformation point, austenite is produced. Consequentl

when the cooling is carried out down to ordinary temperature, the austenite is transformed to the marten which does not undergo the annealing. Thus, the sulfide stress corrosion cracking-resistance is conspicuously deteriorated.

As mentioned above, the high strength seamless steel pipe having excellent sulfide stress corrosion cracking-resistance can be obtained. According to the present invention, in order to further enhance the strength and the souring critical stress ratio, Ti, Nb and V may be added.

Ti, Nb and/or V : not more than 0.1%
Ti, Nb and V all form carbides as in the case with Cr and Mo during the quenching and annealing treatment, and effectively contribute to the enhancement of the quenchability and resistance against the annealingsoftening. However, if the addition amount exceeds 0.1%, the precipitates not only become coarse, but also the sulfide stress corrosion cracking-resistance is inversely deteriorated and further the processability and toughness become lowered. Thus, it is necessary that Ti, Nb and V be added in a rang of not more than0.16 either when singly added or when added in combination.

Although the reason why not only the strength but also the sulfide stress corrosion cracking-resistance a remarkably improved when the conditions according to the present invention are met is not definitely m clear, it is thought as follows:
That is, the sulfide stress corrosion cracks are thought to be a kind of hydrogen brittleness in which hydrogen generated by the corrosion of the steel with an aqueous solution containing hydrogen sulfide penetrates into the steel and gathers at stressconcentrating locations of the inclusions, precipitates, transformations etc. to embrittle the steel.The stressconcentrating locations at which hydrogen gathers a thought to be conspicuously reduced by reducing P and
S, decreasing the inclusions, decreasing transformation density and making the precipitates spherical through annealing at high temperatures of not less than 6800C, and obtaining structure in which the precipitates are finely and uniformly distributed through annealing at high temperatures after not less th: 90% is converted to martensite.

Ordinarily, when the annealing is carried out at high temperatures to attain the above-mentioned low transformation density and spherical precipitates, the strength of the steel becomes lower. According to ingredient combination of the present invention, however, it is considered that the tissues having the precipitates with appropriate profile and distribution can be obtained so that the high strength can be obtained without deteriorating the sulfide stress corrosion cracking-resistance.

Best Mode for Carrying out the Present Invention
A seamless steel pipe hollow material having an ingredient composition shown in Table 1 was hot processed, heated up to the Ac3 transformation point, cooled at the average cooling rate shown in Table and then subjected to an annealing treatment at a temperature given in Table 1, thereby obtaining a product.

The yield strength (Y.S.), the tensile strength (T.S.) and the sulfide stress corrosion crackingresistance o the thus obtained seamless steel pipes were examined, and results thereof are shown together in Table 1.

The sulfide stress corrosion cracking-resistance was evaluated by a test in which a stress being 80% of t yield strength was loaded while a round bar tensile test piece was immersed into an NACE liquid (0.5% acetic acid, and saturated hydrogen sulfide aquoues solution containing 5% of sodium chloride). "o" ma and'xtt mark show non-broken samples and broken samples in the test for 30 days, respectively.

Table1(1)-a
EMI14.1

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Table 1(1)-b

EMI15.1

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EMI17.1

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<tb> 20 <SEP> " <SEP> 10 <SEP> 705 <SEP> 90 <SEP> 109 <SEP> x <SEP> "
<tb> 21 <SEP> " <SEP> 20 <SEP> 705 <SEP> 79 <SEP> 92 <SEP> x <SEP> "
<tb> 22 <SEP> " <SEP> 20 <SEP> 705 <SEP> 82 <SEP> 93 <SEP> x <SEP> "
<tb> 23 <SEP> " <SEP> 10 <SEP> 705 <SEP> 83 <SEP> 94 <SEP> x <SEP> "
<tb> 24 <SEP> " <SEP> 60 <SEP> 685 <SEP> 90 <SEP> 110 <SEP> o <SEP> occurred
<tb> 25 <SEP> " <SEP> 0.8 <SEP> 685 <SEP> 80 <SEP> 102 <SEP> x <SEP> "
<tb> 26 <SEP> " <SEP> 50 <SEP> 680 <SEP> 63 <SEP> 73 <SEP> o <SEP> "
<tb> 27 <SEP> " <SEP> 40 <SEP> 700 <SEP> 65 <SEP> 76 <SEP> o <SEP> "
<tb>

As obvious from the Table, all the seamless steel pipes (Nos. 1-10) obtained according to the present invention exhibit high YS and TS values as well as excellent sulfide stress corrosion cracking-resistance

To the contrary, Comparative steels (Nos. 11 and 12) in which the ingredient composition satisfied the appropriate ranges but the annealing temperature did not reach the lower limit and Comparative steel (N 25) in which the cooling rate during quenching did not reach the lower limit were poor in the sulfide str corrosion cracking-resistance. Comparative
Steel (No. 24) of which the cooling rate during quenching exceeds the upper limit was excellent in the sulfude stress corrosion cracking-resistance, but produced the quenching cracks.

Among the steels (Nos. 13-23, 26, and 27) in which any of the ingredients falls outside of the appropriat range of the present invention, Steels (Nos. 18, 26 and 27) in which C-content was less than the lower li were good in the sulfide stress corrosion cracking-resistance, but gave only the lower YS and TS values The other steels (Nos. 13-17) and 19-23) were excellent in the YS and TS values, but were poor in the sulfide stress corrosion cracking-resistance.

Industrial applicability
According to the present invention, it is possible to obtain the seamless steel pipe which is composed of inexpensive ingredient system containing no great amount of expensive elements unlike the prior art an suitable for the continuous casting and which has the high strength of the yield strength of from 75 to 1 kgf/mm2 and is free from the development of the sulfide stress corrosion cracks under the load stress of 80% of the yielding stress. Thus, the present invention is advantageously suited particularly for the oil v pipes and line pipes for souring gas and souring oil under the tendency that the wells become deeper un the stronger souring and further pipe lines for the chemical plant.

Claims of corresponding document: **EP0224591**

What is claimed is:

1. A method of manufacturing a high strength seamless steel pipe being excellent in sulfide stress corrosion cracking-resistance, which comprises hot processing a hollow raw material for the seamless st pipe containing from over 0.40 to 0.60 wt% of C, from 0.20 to 0.35 wt% of Si, from 0.4 to 1.2wt of Mn, from 0.8 to 1.5 wt% of Cr, from 0.6to 1.0 wt% of Mo, from 0.005 to 0.1 wt% of Al with P being restrai to not more than 0.020 wt% and S being restricted to not more than 0.010 wt%, performing heating at a temperature not lower than Ac3 transformation point, and effecting quenching in which cooling is done the average cooling rate 1 to 500C/s from said temperature to at least 3000C to produce a martensite tissues, and then effecting annealing in a temperature range of not lower than 6800C and not higher thar Ac1 transformation point.